

# News Briefs

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## General Developments

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### ENERGY-RELATED INVENTIONS PROGRAM MAKES RECOMMENDATIONS

During February and March 1997, the NIST Office of Technology Innovation recommended three innovative technologies for commercialization to its Department of Energy partner under the Energy-Related Inventions Program. They include: Laser Ultrasonic Furnace Tube Coke Monitor—a laser-based instrument for measuring the thickness of coke deposits in a tubular industrial furnace while the furnace is in use; The Anaerobic Pump—a new process and hardware design for the anaerobic digestion of organic wastes to produce methane-carbon dioxide (biogas) mixtures; Composite Electrodes for Advanced Electrochemical Applications—a composite electrode for the electrochemical industry using a highly conductive ceramic coating for both the anodes and cathodes.

### U.S.-BRAZIL STANDARDS IN TRADE WORKSHOP, APRIL 7-18, 1997

NIST sponsored a Standards in Trade Workshop April 7-18, 1997, for 20 standards and conformity assessment officials from the government and private sector of Brazil. This successful workshop helped to establish the NIST Regional Standards Attaché, who is stationed with the Commercial Section of the U.S. Embassy in Buenos Aires, Argentina, as an important and knowledgeable technical resource for South American countries; in particular, the attaché serves to facilitate the flow of information on standards and conformity assessment practices between the United States and Brazil.

The workshop familiarized U.S. and Brazilian participants with each other's technology and practices in standardization, conformity assessment, metrology, and measurement systems. Experts from NIST, the American National Standards Institute (ANSI), and other key government and private-sector organizations, as well as Brazilian participants, provided briefings that illustrated similarities and differences between the two standards and conformity assessment systems as well as the roles of the private and governmental sectors in each system. Activities included visits to NIST laboratories, the Department of Commerce, the U.S. Chamber of Commerce, and the American Society for Testing and Materials (ASTM). Working receptions were hosted by ANSI, the National Fire Protection Association, and the Embassy of Brazil. Discussions throughout the workshop focused on the importance of continued trust and mutual confidence to enhance trade.

Brazilian participants proposed next steps for future shared projects, such as research exchanges, the establishment of a Standards Reference Materials Program within Brazil, and a similar Standards in Trade workshop to be hosted by Brazil. It was acknowledged that NAFTA and Mercosul are important building blocks for the formation of the Free Trade Area of the Americas, and that the professional relationships established during the workshop will greatly enhance follow-up and future collaboration.

### VXibus-BASED VOR TEST SYSTEM DEVELOPED

A VXibus-based test set has been developed by NIST scientists that is designed to test VHF omnidirectional ranging (VOR) receivers used in aircraft navigation. VOR transmitters propagate waveforms that consist of a 10 kHz subcarrier, which is frequency- and amplitude-modulated by two 30 Hz signals. VOR receivers demodulate these signals and measure the phase angle between the 30 Hz signals to determine the angle from true north to the transmitter. The term "VXibus" refers

to Institute for Electrical and Electronics Engineers Standard 1155-1992, a specification for describing the backplane interconnection and communications protocol of standard-sized modules, contained in a set of standard-sized enclosures, or crates, for housing and powering the modules.

The new VOR test set, based on another test system originally developed by a team of NIST scientists employs a VXIbus-controlled arbitrary waveform generator and an analog-to-digital converter to synthesize and to sample VOR waveforms, respectively. Control and signal analysis software was developed in a graphical programming language (LabVIEW™) that simplifies the human interface to the VXIbus instruments. The system was developed to support the large inventory of VOR receivers still in use by the Army and to assess the state of VXIbus instrumentation as possible replacements for stand-alone instruments in the Army calibration laboratories and mobile vans.

This new test set also will be used to augment the NIST phase angle calibration services for testing commercial VOR receivers. NIST expects that next year this capability will replace the VOR calibration services previously offered at NIST Boulder.

#### **ON-WAFER NOISE TEMPERATURE MEASUREMENT METHODOLOGY RESPONDS TO INDUSTRY NEEDS**

NIST scientists have developed the theoretical formalism and experimental methods for performing accurate noise-temperature measurements on wafer in response to industry needs. Noise temperature is a measure of the noise power available from a given source.

Characterization of the noise properties at microwave frequencies of microelectronic devices fabricated on wafer is an essential ingredient in the design and testing of components which are used in virtually all modern electronic systems, including those used in communications, home entertainment, radar, and test and measurement. Methods and even commercial systems exist for the noise measurements of most interest on wafer. There is a continuing need to improve and extend all such methods, but NIST has identified some of the more pressing needs to be in the areas of accuracy assessment, traceability, and general quality assurance. The current work constitutes a first step in responding to these needs.

The formalism and measurement methods were verified in a series of tests to measure known noise temperatures on wafer at frequencies around 8 GHz. With known off-wafer noise sources, several different configurations were used to obtain different, known, on-wafer noise temperatures. The noise temperatures produced on wafer ranged from about 160 K to about 7600 K. These were then measured, and the results were compared to predictions. Good agreement was found, with a worst-case disagreement of 2.6 %. An uncertainty analysis of the measurements resulted in a relative standard uncertainty (one standard deviation estimate) of 1.1 % or less for most values of noise temperature. The tests also confirm the group's ability to produce known noise temperatures on wafer. The work is reported in NIST Technical Note 1390, which presents the theoretical formulation and describes the design, methods, and results of the tests performed to verify NIST's ability to measure on-wafer noise temperature.

#### **NIST-SANDIA COLLABORATION ESTABLISHED TO PURSUE SINGLE-CRYSTAL CD ARTIFACT**

Fourteen companies have joined a new NIST consortium intended ultimately to provide industry with a new class of artifacts for critical-dimension (CD) measurements, based on collaborative work at NIST and Sandia National Laboratories. Feature linewidth on these artifacts can be measured using all known techniques including electrical probing, transmitted optical microscopy, scanning electron microscopy (SEM), transmission electron microscopy, and scanned-probe microscopy. The line features of the artifacts, fabricated at Sandia of single-crystal (110) or (100) silicon, have planar sidewalls, either vertical with respect to the plane of the substrate or having a known slope angle of approximately 57°, respectively.

The intense interest in this development lies in the anticipation that the fabrication methodology will lead to a critical-dimension artifact standard from NIST, traceable to the meter, having deep-submicrometer feature widths. At present, companies rely on various methods for CD measurement, with scanning-electron microscopy being dominant. Many companies have developed internal methods for standardizing the measurements of one or more specific procedures.

These measurements are not calibrated, in the sense that they are not traceable to the meter; further, there is generally little correlation among the results of various measurement methods. An additional factor is that edge-determination issues for CDs at or below the 180 nm level are likely to cause industry to seek tools other than the SEM for primary CD measurement.

Through the establishment of the collaboration, NIST and Sandia are responding to the need for a manufacturable structure having known geometric properties, specifically, narrow lines of rectangular or otherwise known cross section, having sidewalls defined by the method. Difficulties encountered in earlier attempts to fabricate by conventional methods a “universal” artifact are reduced greatly because ambiguities in the width of the line have been eliminated. The work also is expected to lead to improvements in other electrical test structures widely used in industry, including those developed at NIST.

#### **NIST WORK UNDERLIES IEEE GUIDE FOR MEASUREMENT OF QUASI-STATIC MAGNETIC AND ELECTRIC FIELDS**

The Institute of Electrical and Electronics Engineers (IEEE) has published Standard 644-1996, “IEEE Guide for the Measurement of Quasi-Static Magnetic and Electric Fields.” This action represents the culmination of work begun at NIST 2 years ago in collaboration with the ELF (Extremely Low Frequency) Field Measurements Working Group, a component of Subcommittee 1 of IEEE Standards Coordinating Committee 28. The first draft and subsequent revisions were prepared at NIST on behalf of the working group. The guide is intended to aid groups and individuals interested in developing magnetic and/or electric field measurement protocols by describing different magnetic and electric field measurement methods that can accomplish specific measurement goals. A single measurement approach is not identified because the measurement strategies and instrumentation requirements will differ depending on the measurement environment of interest and the goals of a measurement program. For example, the measurement protocols and instrumentation for characterizing electric and magnetic fields from power lines will differ significantly from those for characterizing fields from electrical appliances such as hair dryers and shavers.

A companion document for this Guide is IEEE Standard 1308-1994, “Recommended Practice for Instrumentation: Specifications for Magnetic Flux Density and Electric Field Strength Meters-10 Hz to 3 kHz,” which describes the types of available instrumentation used for measuring quasi-static magnetic and electric fields, their principles of operation, definitions of terminology, calibration procedures, and sources of measurement uncertainty. Frequent reference is made in the Guide to IEEE Standard 1308. Drafts of IEEE Standard 1308 also were prepared at NIST, in collaboration with the AC Fields Working Group in the IEEE Power Engineering Society.

#### **IEEE PES RECOGNIZES DEVELOPMENT OF HIGH-VOLTAGE TESTING STANDARD INCORPORATING NIST CONTRIBUTIONS**

The Institute of Electrical and Electronics Engineers (IEEE) Power Engineering Society (PES) has named the High Voltage Test Techniques (HVTT) Subcommittee to receive its 1997 Working Group Recognition Award for the publication of the revision to IEEE Standard 4, “Standard Techniques for High Voltage Testing.” The PES recognizes its working groups with two awards given annually: one for the best technical report and the other for the best standard or guide. NIST has provided significant technical support to the development of this revision of Standard 4, particularly in the sections covering the measurement of impulse high voltages. A convolution technique developed at NIST has been incorporated into Standard 4 as one of the tests for qualifying high-voltage impulse dividers. This technique checks the temporal response of dividers with a low-voltage step pulse to determine if they are suitable for measuring impulses having microsecond risetimes. The digitized response is mathematically convolved with an analytic waveform of the type to be measured. A comparison of the resultant waveform to the analytic input waveform then shows if the divider introduces significant measurement errors.

The latest revision of Standard 4 is the seventh edition of a document that has existed as a separate standard since 1928. (Historical note: The subject of high-voltage measurements was addressed in the earliest Standardization Report of the Institute of Electrical and Electronics Engineers (AIEE) in 1889.) The seventh edition incorporates major changes from

the sixth, including new tests such as the convolution test described above. In addition to the PES Working Group Recognition Award, HVTT's development of the new edition was recognized in 1996 by the IEEE Power Systems Instrumentation and Measurement (PSIM) Committee for "producing the best standard of the previous three years."

### HEXAPOD MACHINE TOOLS

Hexapod machine tools have a number of potential advantages over conventional machine tools. These include high stiffness, low weight, low effective inertia of the moving components, and mechanical simplicity. Disadvantages include complex workplace, a small orientation range, and, unless measures are taken, low structural damping. A NIST researcher has completed a series of experiments to assess the errors of the prototype hexapod milling machine located at NIST. His results indicate that the Hexapod errors differ from those of conventional machine tools. Since each strut only realizes a distance, its roll, pitch, yaw and straightness errors are not important. However, new errors are introduced. These errors stem from the positions of the platform and base joints, the accuracy of these joints, and the average length of the struts.

In an unloaded state, the geometric errors are determined by 35 constant parameters and 18 functions. Errors in the relative strut length can be measured individually. The significant constant error parameters were estimated simultaneously by using conventional performance evaluation metrology. However, there is no straightforward relationship between the results of such tests and the parametric errors. More optimized error assessment procedures are needed, including those focused on subsets of the error parameters.

The data also indicate a need for closed-loop thermally invariant metrology that addresses the length of the total strut, low-friction high-stiffness joints, and a higher structural damping.

### HEXAPOD USER GROUP MEETING

The National Advanced Manufacturing Testbed (NAMT) hexapod project team hosted a meeting of the Hexapod User Group on March 26, 1997, at NIST. The Hexapod User Group is an informal organization of machine tool builders who are building Stewart

platform-based hexapod machines, government and university engineers who are conducting hexapod machine research, and machine tool users who are following the development of these machines with an eye toward adopting the technology for use in their production operations. More than 40 people attended the meeting, with approximately equal representation from each of these three categories. This was the second meeting of the group; the first was held at MIT in August 1996.

The goals of the meeting were to:

- provide a forum for discussion of hexapod machine tool research issues;
- obtain a status update on the different hexapod research and development efforts in progress; and
- serve as a catalyst for identifying potential collaborative projects and leveraging opportunities.

The meeting featured 10 presentations on topics such as the state of commercial machine development, hexapod calibration, error modeling and assessment, workspace analysis and approaches to modeling and simulation, and control algorithm development. Several "focus areas" such as hexapod metrology, applications, machining test parts, and art-to-part approaches also were discussed. One machine tool maker presented test results indicating impressive progress toward obtaining performance competitive with conventional serial/Cartesian machines. NIST scientists shared thermal characterization data recorded on the octahedral hexapod machine at NIST; reported considerable success in improving the performance of the machine through calibration; demonstrated the NIST octahedral hexapod machine; and showed hexapod modeling, simulation, and control system capabilities being developed as part of the NAMT.

The meeting provided representatives of machine tool users with an opportunity to discover the current status of the technology—"a font of information" in the words of one industry attendee, as well as to express views on the direction of hexapod technology development. Two participants announced future meetings of interest: a special panel session on hexapod machines at the ASME International Mechanical Engineering Congress and Exhibition to be held Nov. 19-21, 1997 in Dallas, TX; and a first meeting of the newly formed European Hexapod User Group to be held in Nottingham, England, this fall.

### ATDNET DEMONSTRATION OF NAMT

The National Advanced Manufacturing Technology (NAMT) program successfully conducted its first experiment on ATDnet, a Washington-Area, high-speed Asynchronous Transfer Mode (ATM) network connecting several government agencies. NAMT participated in an ATDnet capabilities demonstration at the recent FOSE conference in Washington DC. Live, full-motion video was sent from several NAMT project sites at NIST, to the Federal Office Systems Expo (FOSE) conference, through the ATDnet. FOSE attendees also were able to manipulate the pan/tilt/zoom cameras, at NIST, from a workstation at FOSE. The importance of this experiment was to show how ATM networks can be used to transport simultaneously data, voice, and full-motion video across a wide geographic area.

### SIMULATING THE SPATIAL EVOLUTION OF CHEMICAL REACTIONS AT BIOMOLECULAR SURFACES

Molecular recognition in biology and in bioanalytical applications involves complex molecular shapes, reactive surfaces, and diffusion processes. Diffusion limited reactions involving macromolecules can be described by Brownian motion and the Smoluchowski kinetics model that requires the computation of the electrostatic capacitance of an irregularly shaped object to describe the bimolecular chemical reaction rate of small molecules with complex macromolecules.

An algorithm for calculating these rates recently has been developed at NIST. This algorithm uses a small library of exact Green's functions for the Laplace equation to eliminate the need to explicitly construct those portions of a diffusing particle's trajectory that are not near an absorbing surface. This computational procedure virtually eliminates systematic sources of error in the calculation of capacitance; therefore, it considerably simplifies the construction of computer simulations of these types of problems. This treatment is especially efficient when the reactive objects have highly irregular shapes and, as such, will be an extremely important tool for modeling reactions involving biological macromolecules. A model based on this algorithm can address questions such as how the density of reactive sites at a surface influences the total reactivity of the surface. For example, a spherical surface containing totally reactive disc-shaped sites that occupy 20 % of the total surface area is predicted to be as reactive as another surface covered with 100 % such sites. The model also allows accurate predictions of how the geometry of the reactive sites and their relative locations influence the overall surface reactivity. Continued development of the model will permit

detailed predictions concerning the evolution of size and shape of self-organizing groups of molecules at reactive surfaces.

### ELLIPSOMETRY OF PROTEIN ADSORPTION ON METALS

Ellipsometry is a well-established optical method for measuring the extent and kinetics of organic molecule adsorption on metal surfaces. Interpretation of the ellipsometric measurements to acquire parameters of the adsorbed phase such as its thickness and its complex index of refraction requires an accurate optical model of the interface. This interface is commonly described in terms of three phases—bulk metal/adsorbate film/solution. The ellipsometric contributions in a stratified media are additive. NIST scientists have shown that protein film properties for ellipsometry measurements of a ferredoxin adsorbing on gold are poorly represented by the three phase model over the range of 400 nm to 750 nm (spectroellipsometry). The problem is due to the model's failure to account for the interfacial charge redistribution which makes the protein film look "gold-like" in the three phase model. A quantitative method that accounts for the influence of the metal surface on the optical properties of the adsorbing protein film has been developed. Simultaneous measurements of electrode impedance and in situ spectroscopic ellipsometry allow the parameters of the model to be specified. This leads to the elucidation of the complex index of refraction spectrum for the adsorbed protein that is free of the influence of the substrate's optical properties. This index of refraction can be used for calculating protein surface concentration as a function of adsorption time, which is of practical value to biomaterials research and manufacture. The results of this study will appear in the journal *Langmuir*.

### NEW MICROCONTAMINATION MODEL FOR CHEMICAL VAPOR DEPOSITION REACTORS

Contamination via small gas-phase generated particles, i.e., microcontaminants, is a major problem afflicting semiconductor processing in chemical vapor deposition (CVD) reactors. Researchers at NIST have developed a new model for analyzing this problem in the commonly used rotating disk CVD reactor. This model utilizes a flow/chemistry/aerosol formulation to predict the locations where particles form as well as the subsequent evolution and transport of these particles. Of particular concern is the rate at which the largest of the micro-contaminants, i.e., the "killer" particles, impact the fabricated material and thus render it defective. The

model generates performance maps, which indicate both material growth rate and contamination rate as functions of reactor operating parameters. A set of parameters then can be selected, which results in an optimal growth rate without excessive contamination. A planned series of experiments at NIST will be used to validate the predictions of this unique new tool for enhancing the productivity of the semiconductor fabrication process.

#### NEW STEAM TABLES IN SOFTWARE FORM

NIST scientists in cooperation with the American Society of Mechanical Engineers, have produced a new software program for the calculation of the thermophysical properties of water. The program is distributed by NIST's Standard Reference Data Program as Standard Reference Database 10: NIST/ASME Steam Properties.

Engineers long have used steam tables in the power industry for the design of chemical plants and refineries and in many other applications. Accurate water properties also are needed for solution chemistry and other research. Standards for water properties are set by the International Association for the Properties of Water and Steam (IAPWS). IAPWS recently adopted a new formulation for the thermodynamic properties of water. The new NIST software implements this formulation, along with other present and proposed IAPWS standards for transport properties, surface tension, the dielectric constant, and fluid/solid phase boundaries. Calculations may be performed along saturation boundaries, along constant-property paths such as isotherms, and at specified states of two independent variables. The user can specify which properties to display and the units in which to enter and display data. Additional features of the software include on-line help, plotting capability, and the ability to copy and paste data to and from other applications.

A key feature of the new NIST program is that the underlying calculations are modular, via well-documented interface routines. This makes it easy for others to integrate the calculations into their own programs. NIST recently concluded an agreement with a private company, to allow the company to integrate NIST's calculational routines in their widely used chemical process simulation software.

#### PREVIOUSLY UNKNOWN SOURCE OF ERROR IN CHEMICAL ANALYSIS IDENTIFIED AND CORRECTED

Research conducted at NIST has identified a previously unrecognized source of error in chemical analysis performed using glow discharge optical emission spectrometry (GDOES). GDOES has been used for

many years in industrial labs for the elemental analysis of materials directly in the solid state. Although the technique has been applied traditionally to the analysis of electrically conductive materials, such as metals and alloys, its applicability recently has been extended to electrically nonconductive materials, such as ceramics, glasses and polymers as well as to the depth-resolved analysis of films and coatings.

Despite the broad applicability and long-time use of the technique, the optical systems used to measure emission from the glow discharge device have in some respects remained in a state of developmental infancy. Specifically, emission from the device is usually transferred to the spectrometer via a single lens, which images the discharge onto the entrance slit. The suitability of this simple-minded approach for application in analytical GDOES has met with little, if any, scrutiny.

In a recent paper, it was reported that material removed from the sample surface during analysis did not mix thoroughly in the gas phase in their glow discharge device. This suggests that the use of a single lens to image the discharge onto the entrance slit may induce analytical error when the sample is chemically heterogeneous. During the NIST studies, the behavior of GDOES with samples known to be chemically heterogeneous was evaluated in order to investigate this possibility. Two samples were used. The first was a synthetic sample consisting of a brass surface with a 1 mm diameter implanted steel plug. Since this sample is not realistic in terms of its severity of heterogeneity, a second sample also was employed. This sample was a brass disc that had been rejected previously as a standard reference material, owing to a small Pb heterogeneity.

Studies with both samples indicated the presence of optically induced analytical error. The error demonstrated for the synthetic sample was about 35 %, indicating that the error can be large. In two trials, the error associated with the more realistic sample was between 1 % and 2 %. This magnitude of error is of concern in quantitative GDOES analyses.

Elimination of the optically induced error was attempted with the use of an alternative optical transfer method. This method, which essentially averages over the entire volume of the glow discharge, utilizes two lenses to transfer emission from the discharge device to the entrance slit. The alternative approach successfully eliminated the error for both the synthetic and "real-world" samples, while maintaining analytical sensitivity and low limits of detection. These results suggest that the two-lens system should be employed routinely in analytical GDOES. This work recently was published in *Appl. Spectrosc.* 50(2), 245 (1996).

### NIST SCIENTISTS HOLD WORKSHOP ON NIST-NIH DESKTOP SPECTRUM ANALYZER

NIST research staff members hosted 26 scientists and engineers from industry, university, national laboratories, and other U.S. government laboratories at a special workshop on the NIST-NIH Desktop Spectrum Analyzer (DTSA) March 26-28, 1997. More than 200 scientists have attended these workshops since their inception. DTSA is a comprehensive x-ray spectrometry software engine developed by NIST and NIH scientists for application to electron beam instrumentation, including the scanning electron microscope, the electron probe x-ray microanalyzer, and the analytical electron microscope. DTSA is capable of spectral collection, peak deconvolution from spectral overlaps and background, qualitative analysis, quantitative analysis through detailed matrix corrections, and spectrum modeling from first principles at all stages of generation, propagation, and detection. Incorporated into DTSA is a complete database of x-ray information, which forms the basis for the advanced mathematical procedures for peak deconvolution. DTSA is capable of processing spectra measured with the semiconductor energy dispersive x-ray spectrometer, the wavelength dispersive (crystal diffractometer) spectrometer, and the new NIST microcalorimeter energy dispersive x-ray spectrometer.

### NEW X-RAY DATABASES AVAILABLE ONLINE

Information developed for use by those working in x-ray diffraction and related fields has been converted into a World Wide Web (WWW) database and is now available online: the *Theoretical Form Factor, Attenuation, and Scattering Tabulation for  $Z = 1-92$  from  $E = 1-10$  eV to  $E = 0.4-1.0$  MeV* (FFAST). These data have been available previously in printed and magnetic media formats, but the WWW database presents the information on demand in both graphical and tabular form.

Information is provided for each atom at each of many energies. It includes the total attenuation coefficient, the photoelectric cross section, the sum of the coherent and incoherent scattering, and self-consistent values of the atomic scattering factors  $f_1$  and  $f_2$ . The WWW user of this database is given significant control over the information that is viewed. Those using a web browser with a frames feature can have both tabular and graphical information delivered on the same screen. (Browsers not supporting frames receive the same information sequentially.)

The FFAST database may be accessed on the WWW at the URL: <http://physics.nist.gov/PhysRefData/FFast/Text/cover.html>. A complete set of the databases currently provided by the Physics Laboratory's Office of Electronic Commerce in Scientific and Engineering Data may be accessed at the URL: <http://physics.nist.gov/PhysRefData/contents.html>.

### SENSITIVE DIAGNOSTICS OF INFRARED FILTERS

Narrow-band infrared filters are devices that are transparent only over a small range of infrared wavelengths. They are used in applications such as two-color optical pyrometers (to remotely measure the temperature of hot objects), filter radiometers (such as those used to calibrate multispectral imagers), and military guidance and infrared seeker systems. Small "leakage" bands on the order of  $10^{-5}$  to  $10^{-4}$  in transmittance are often present in these filters in the wavelength regions where they are nominally opaque. This leakage can be of critical importance in some applications where an infrared sensor must be very well shielded from unwanted radiation.

NIST has developed the capability to measure the out-of-band rejection of narrow-band infrared transmittance filters down to a transmittance level of  $10^{-6}$ , over a wavelength range from 2  $\mu\text{m}$  to 20  $\mu\text{m}$ , at temperatures from 10 K to 300 K. To allow the out-of-band transmittance to be measured with high sensitivity, the measurements are performed using a Fourier transform infrared spectrometer and a set of high- and low-pass optical filters to block the main band transmittance of the filters under test.

### NEW 2.5 METER PHOTOMETRIC INTEGRATING SPHERE

A new 2.5 m integrating sphere has been installed at NIST to improve flux measurement standards for a variety of lamp types, such as incandescent, fluorescent, and discharge. This new sphere will be used for the NIST realization of the SI unit of luminous flux, the lumen, and for improving photometric calibration services using a new detector-based measurement method.

The lumen is a measure of the total light from a lamp, in all directions, as evaluated using the human eye response. It is the radiometric quantity of most interest for lamp manufacturers and others in the lighting industry. The large sphere reduces the uncertainties associated with the geometrical size and shape of the test lamps and with the stability of ambient temperature for fluorescent lamps.

NIST recently developed a new method for realizing the lumen using an integrating sphere rather than the traditional goniophotometer. The new 2.5 m sphere will be equipped with additional optical instrumentation to allow a world's first: the detector-based calibration of luminous flux. The new method will result in a shorter calibration chain and reduced uncertainties in luminous flux calibrations.

### MEDICAL IMAGING WITH POLARIZED $^3\text{He}$

Starting in 1992, researchers at NIST have been developing an ability to produce large quantities of polarized  $^3\text{He}$  gas. In ordinary helium gas, the nuclear spins of the atoms point in random directions; however, in a polarized sample, the nuclear spins are made to point preferentially in a given direction.

The initial reason for developing this competence was to produce a neutron polarizer based on preferential transmission of one spin state of an unpolarized neutron beam through the polarized gas. However, recent events have shown that this technology has another, previously unexpected application: magnetic resonance imaging (MRI) for medical diagnosis. While ordinary MRI techniques rely on the proton spin in water molecules, researchers have found that it is possible to obtain MRI pictures by using polarized  $^3\text{He}$  or  $^{129}\text{Xe}$  gas as a contrast agent. For example, if a patient inhales one of these inert gases, the lungs can be imaged. (Normally lung tissue cannot be imaged with MRI because of its low density and low water content.)

Researchers at NIST are collaborating with researchers at the Metabolic Magnetic Research and Computing Center at the University of Pennsylvania to develop polarized  $^3\text{He}$  MRI. In this collaboration, NIST produces the cells filled with gas and shares its expertise in how to produce highly polarized gases using optical pumping. The Pennsylvania collaborators contribute their knowledge in MRI techniques and the MRI apparatus. The project recently passed a significant milestone by producing a high-resolution image of a set of human lungs using this process. Work continues to improve the technology and develop techniques useful for clinical applications.

### NIST FINDINGS LEAD TO HIGHER PRODUCTIVITY IN CERAMIC GRINDING

Mechanical components made of advanced ceramics frequently offer significant advantages over other materials in terms of improved wear resistance, resistance to corrosion, and capability to operate at elevated temperatures. High manufacturing costs, however, have

impeded the extensive use of these materials. By some estimates 50 % to 80 % of the cost is associated with machining. Grinding with diamond grit wheels is the predominant method used for shaping and final finishing. Because of potential surface and sub-surface damage in the form of microcracks and a resulting increased risk for part failure, the grinding process is generally carried out at low material removal rates.

In conjunction with the NIST Ceramic Machining Consortium, the effect on strength for a range of different grinding conditions has been obtained for three types of silicon nitride ceramics. Utilizing an experimental design that was prepared by NIST scientists, the optimum conditions were determined for grinding the ceramics at high material removal rates. The results of this study have shown that a high specific material removal rate does not necessarily lead to an increased loss in strength. By utilizing the data obtained in this study, it is anticipated that increased efficiency and reduced ceramic manufacturing costs can be achieved.

### NIST ELECTRONIC PACKAGING PAPER RECEIVES AWARD

A recent award-winning paper describes work from the NIST program on microelectronic packaging that addresses an important technical challenge facing the microelectronics industry: how to measure reliably the relevant properties of exceedingly thin polymer films and highly filled polymers materials. Industry needs these measurements to enhance the robustness of multicomponent material interfaces and to improve the resistance of plastic assemblies to effects of moisture. A suite of advanced measurement techniques that includes neutron and x-ray reflectivity together with solid state nuclear magnetic resonance spectroscopy (NMR) were used in the NIST work to determine quantitatively the distribution of water and hygroscopic expansion of extremely thin polymer films. The neutron and x-ray reflectivity techniques also measure directly the coefficient of thermal expansion of thin polymer films and neutron techniques yielded information about the concentration profile of water near an interface. NMR measurements of water uptake in a commercial epoxy molding compound revealed that liquid-like water does not accumulate in the mold compound under high-humidity storage conditions and hence is not a contributor to "popcorning," which can occur during solder reflow. However, liquid-like water does accumulate during aggressive accelerated conditioning leading to a mold compound, which is not representative of that found in an electronic package subjected to high storage humidities.



**AUTOMOTIVE METAL FORMING PROGRAM**

Sheet metal forming, always an important element of automobile manufacture, has become critical now that the industry is attempting to switch to high strength steels and aluminum alloys to reduce vehicle weight and hence to improve fuel economy. The behavior of these materials during stamping is significantly different from that of the currently used mild steels, and the major U.S. automobile companies and their suppliers are, therefore, focusing attention on the efficient forming of these new materials. NIST has been involved in this activity through collaboration in ongoing external projects.

Finite-element modeling (FEM) is at the core of modern automotive metal forming operations. It has replaced the earlier, more empirical approach, but considerable trial and error is still required, largely because of a lack of suitable materials data and because of shortcomings in the models themselves. In the former case, many different forming tests are employed by industry, but none provide data of the type required for FEM. A major component of the NIST effort is the development of a relatively small number of standard tests to provide the necessary data. The major shortcoming of existing finite-element models is that they are limited by poor basic understanding of the mechanical property changes that occur during forming operations. The second part of the NIST program is designed to significantly increase fundamental understanding through the use of sophisticated new measurement techniques developed by NIST scientists.

NIST has consolidated its overall program and also added an important new element, namely information transfer between NIST and remote forming facilities. A prototypical forming operation, located at the University of Michigan, will be data-linked to computers at NIST performing modeling using an improved finite-element code developed in collaboration with a private corporation. An instrumented plane strain tensile test will be developed at NIST, which will generate appropriate data for entry into the process model and for transfer to the forming facility. Three-dimensional shapes of the prototypical parts will be measured on-line using techniques developed by NIST, and these shapes will be compared with those predicted by FEM.

**NEUTRON GUIDE NETWORK COMPLETED AT THE COLD NEUTRON RESEARCH FACILITY**

The installation of the new supermirror cold neutron guide network, which delivers beams to the Cold Neutron Research Facility (CNRF) at the NIST Center for Neutron Research, is essentially completed, thus

providing U.S. science and technology with the benefits of the world's largest area of cold neutron beams. Constructed of highly polished glass with reflective, metallic coatings, neutron guides transport long-wavelength neutrons over long distances in a manner analogous to light transport by fiber optic cables: all neutrons with an angular divergence less than the critical angle for total reflection for the guide surface are reflected back into the guide cavity. Using technology developed and tested with the help of NIST scientists, the CNRF neutron guides use  $^{58}\text{Ni}$  coated mirrors, or  $\text{NiTi}$  multilayer coatings called supermirrors, to increase the critical reflection angle and thus provide almost twice the usable neutron flux of natural Ni-coated mirrors. Eight guide tubes made from these neutron mirrors transport neutrons from the new liquid hydrogen moderator over distances of nearly 70 m to 15 state-of-the-art cold neutron spectrometers in the NIST CNRF. The guides are carefully aligned by NIST technicians with an accuracy better than 20'' over their entire length and then sealed and evacuated to minimize transport losses.

Already the only facility of its kind in the United States, the completed guide network will have the largest total area of cold neutron guide available anywhere in the world. The size and intensity of the beams greatly enhance measurement capabilities using cold neutrons for many applications in materials research, chemistry, physics, and biology. NIST operates the cold neutron spectrometers in the CNRF Guide Hall as a national user facility providing unique measurement capabilities to the entire U.S. scientific community.

**1997 AWARD FOR EXCELLENCE IN TECHNOLOGY TRANSFER PRESENTATION**

Two mechanical engineers at NIST have received a 1997 Award for Excellence in Technology Transfer from the Federal Laboratory Consortium (FLC). The award recognizes the engineers' work to develop, patent, and transfer to the public technology for using a solar photovoltaic modules to electrically heat stored water via multiple in-tank resistive elements and a system control module. The system maintains the annual conversion efficiency of the energy generating system at approximately 95 % of the theoretical maximum and avoids the need for expensive auxiliary equipment such as a battery storage system, special direct current (dc) appliances, a dc to ac power inverter, load matching electronics, and/or a two-way utility electrical energy meter. As compared to solar water heating systems that are currently in use, the photovoltaic system is much more reliable, is expected to have a longer operating life, is well suited for all regions of the country, and is more

adaptive to a dual-use function. The technology is being transferred by three instrumented field demonstration sites, conference and small group presentations, journal papers, press releases, magazine articles, regional television, and web pages, the sum of which have led to numerous inquiries and the mailing of more than 500 information packages to interested parties.

The Federal Laboratory Consortium represents over 500 federal laboratories that contribute to the missions of 14 Federal agencies. Each year, nominations for the Excellence in Technology Transfer Awards are selected by a committee composed of members from industry, state and local government, academia, and federal laboratories. The NIST engineers, who were among 30 winners for 1997, were presented a commemorative plaque and medallion during a banquet at FLC's April 14-17 National Technology Transfer Conference.

#### **INNOVATIVE ANTENNA CALIBRATION ALGORITHM AND SOFTWARE**

An algorithm and corresponding software have been developed at NIST for the processing of antenna measurements corrupted by probe position errors. The method exploits position information available during the measurement procedure to compute far fields as accurately as when no position errors are present, at a computational cost, which is acceptable even for electrically very large antennas.

The interpretation of near-field antenna measurements, which requires transformation to the far field, is typically accomplished with the fast Fourier transform (FFT). When the measurement positions deviate from an ideal rectangular grid, however, the FFT is not applicable without modification. The new algorithm employs a combination of a recently developed, unequally spaced FFT, interpolation, and the conjugate gradient method to accurately transform to the far field at a cost proportional to  $N \log N$ , where  $N$  is the number of measurements (typically between 10 000 and 1 000 000).

The method will be used for measurements at higher frequencies and those taken on mobile platforms, where tight tolerances are difficult to maintain. The software is available to antenna measurement laboratories in government and industry and will support the future deployment of communications satellites operating near the terahertz band.

#### **NIST INSTRUMENTAL IN DEVELOPING STANDARD FOR THE EXCHANGE OF FORENSIC INFORMATION**

NIST's successful collaborations with industry and with the law enforcement community resulted in the development of a specification, which supplements a previous standard, for the exchange of forensic information. Consensus on the specification was achieved through a canvass that NIST conducted under its accreditation by the American National Standards Institute (ANSI) as a sponsor of standards for information interchange. NIST also sponsored workshops where participants reached agreements on technical details. The ANSI Board of Standards Review recently approved the Data Format for the Interchange of Fingerprint, Facial and SMT Information (ANSI/NIST-ITL 1a-1997) as an American National Standard. This work is also a component of the framework that NIST has been discussing with the Office of Law Enforcement Standards to support digital representation and exchange of cartridge and bullet imagery data gathered at a crime scene.

#### **ADVANCED SCHOOL FOR METROLOGY: EVALUATION OF UNCERTAINTY IN MEASUREMENT**

The National Institute for Metrology, Standardization and Industrial Quality (INMETRO); the CAPES Foundation; the National Research Council of Brazil; and the Brazilian Society of Metrology jointly sponsored a metrology conference in Brazil recently at which NIST scientists spoke on "Evaluation of Uncertainty in Scientific Measurement," presented a paper on "Design and Analysis of Mixture Experiments," and led a roundtable discussion of the "Impact of Metrology in International Development." They also moderated sessions on practical applications of metrology. The conference attracted 120 people, including speakers from nine nations.

#### **NIST SEEKS TO DEVELOP AN ADVANCED ENCRYPTION STANDARD (AES)**

NIST is seeking a strong cryptoalgorithm that could be used by both the public and private sectors in protecting sensitive unclassified information for the next 20 to 30 years. Other NIST goals for the AES include an algorithm that would support standard codebook modes

of encryption, one that is significantly more efficient than triple Data Encryption Standard (DES<sup>3</sup>), one with a variable key size so that security could be increased when needed, and a selection process that is fair and open.

A *Federal Register* notice of Jan. 2, 1997 announced NIST's intent to develop an AES and proposed minimum acceptability requirements and evaluation factors. Draft submission requirements for candidate algorithms were also announced. A call for comments produced 33 comments for discussion at an April 15 workshop, which NIST hosted on developing an AES. About 80 participants from industry, government, and academia attended the workshop, including representatives from Canada, the United Kingdom, Belgium, and Japan. The discussion focused on the draft minimum acceptability requirements, evaluation factors, submission requirements, intellectual property issues, and the selection process.

NIST intends to issue a public call for submission of candidate algorithms. After the call period closes, NIST will make all submissions available for public review and analysis. Through a series of open workshops and public review periods, NIST will select the best algorithm for the AES based on its ability to provide the required level of security first, then on cost and flexibility considerations. For more information, visit the Web site at <http://csrc.nist.gov/encryption>.

### CONSORTIUM TO PROTOTYPE THE VIRTUAL WAY

NIST has launched a new consortium that aims to increase prototyping capabilities and cut the time required to introduce new parts and products. The goal of NIST manufacturing researchers and their prospective partners is to develop the basis for virtual machine tools and inspection machines that behave just like their factory floor counterparts. "This project will help bridge industry's communication gap between design and manufacturing," says a member of the senior technical staff in producibility engineering at a consortium partner.

By means of NIST's recently opened National Advanced Manufacturing Testbed—a distributed research facility built on a state-of-the-art computing and communications infrastructure—the partners will develop software tools to accurately predict machine performance and, therefore, whether new parts can be machined to as-designed specifications. Examples of such tools include computer models that represent actual machine behavior, mathematical representations of part geometry, virtual machining and inspection algorithms, standardized data formats, and remotely accessible performance-data repositories.

For information on the Consortium for Machine Tool Performance Models and Machine Data Repository, contact Automated Production Technology Division, B108 Sound Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-6600, <[donald.blomquist@nist.gov](mailto:donald.blomquist@nist.gov)>.

### PUBLICATION FEATURES TESTS FOR COMPUTER ACCURACY

If a wristwatch isn't keeping proper time or a thermometer is off by a couple of degrees, you can tell by comparing the errant device to one that's more accurate. But making sure computer programs are up to par is a little trickier.

That is why NIST is working on tests for software and other information technology products and systems. Such research, which encourages companies to develop quality products, is among the highlights of the new publication, *Information Technology Laboratory Technical Accomplishments 1996*.

As a resource that highlights the technical research, industry collaborations and standards-related work of the Information Technology Laboratory, this publication touches on issues emerging from today's information revolution. It covers such topics as human-machine interface technologies, software diagnostics and performance, tests for new standards, computational science and computer security.

NIST's information technology research concentrates on developing tests and test methods for information technologies that are still in the early stages of development.

The information in this publication is available on the World Wide Web at <<http://www.nist.gov/itl>>. For a printed copy, send a self-addressed mailing label to Elizabeth Lennon, A216 Technology Building, NIST, Gaithersburg, MD 20899-0001, or e-mail your address as it would appear on a mailing label to <[elizabeth.lennon@nist.gov](mailto:elizabeth.lennon@nist.gov)>.

### BROCHURE HELPS U.S. FIRMS OBTAIN CE MARK

The "CE" mark is now mandatory for a wide range of products sold in the European Union. It indicates that a product conforms to EU safety, health and environmental legal mandates. The European Commission calls it "a passport" that allows manufacturers to trade industrial products freely within its internal market. Unfortunately, many U.S. manufacturers view the process of securing a CE mark as difficult and time consuming.

A new brochure "CE Alert," prepared by NIST, ends the confusion surrounding the CE requirement and

makes it easier for American businesses to secure the mark. The brochure gives solid recommendations for manufacturers to follow and then works through an example. It also lists sources of information that can provide assistance throughout the process.

Copies of the brochure may be obtained by sending a self-addressed mailing label to the National Center for Standards and Certification Information, Building 820, Room 164, NIST, Gaithersburg, MD 20899-0001. Copies also may be requested by phone: (301) 975-4040, fax: (301) 926-1559, or e-mail: <ncsci@nist.gov>.

### MSTQ SYSTEMS IN 70 NATIONS PROFILED

For the first time officials in industry and government have a single source for obtaining important information on the national metrology, standards, testing, and quality institutions (MSTQ) in more than 70 countries. NIST Special Publication 912, *Profiles of National Standards-Related Activities*, provides users with concise information on the MSTQ systems of the world's major economies and many less-developed countries.

The new directory was prepared for the NIST Office of Standards Services, which serves as the national focal point for information on domestic and international standards and certification programs. A NIST spokesperson notes that in today's global economy, U.S. industry and government agencies must be aware not only of a foreign nation's standards but also have information on the availability and the authority for related activities such as metrology services, conformity assessment activities and institutions, and accreditation bodies.

The new directory is a single resource for obtaining the names and addresses of a country's metrology center and legal metrology agency; its primary standards and quality organizations; government and private-sector accreditors of calibration and testing laboratories; product certifiers, quality and environmental management system registrars; information sources and many other aspects of the country's industrial infrastructure.

Included in each nation's profile are: basic data on its economy and trade, its MSTQ framework, responsible agencies and institutions, and key contacts and information sources. The directory is expected to be a useful reference for all organizations involved in standards and conformity assessment-related activities in the global marketplace.

Copies of SP 912 are available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402, (202) 512-1800. Order by stock no. 003-003-03462-2.

### REPORT EXAMINES FIRE PROTECTION FOR HIGH-CEILING SPACES

A new report by a team of fire prevention professionals from NIST, the Naval Facilities Engineering Command, and private industry should prove useful to those designing, installing or retrofitting smoke detectors, automatic sprinklers and other fire protection systems into high-ceiling structures, such as aircraft hangars, warehouses and hotel atriums.

The team's report, *Analysis of High-Bay Hangar Facilities for Fire Detection Sensitivity and Placement* (NIST Technical Note 1423), looks at placement spacings for ceiling heat detectors, describes methods to improve the effectiveness of projected beam or optical smoke detectors, and compares various sprinkler systems. The researchers gathered data from 33 full-scale fire tests conducted in high-bay aircraft hangars located at naval air stations in Hawaii and Iceland.

The fires, ranging from 0.09 m<sup>2</sup> to 20.9 m<sup>2</sup> in area, were studied in the hangars under varying conditions (including air flow, ambient temperature, fuel type and closed/open doors). Each fire yielded data on 200 separate measurement points.

As a result of these tests, the researchers gained insight into heat and smoke behavior in high-ceiling structures, the response time of fire detection and sprinkler systems, the ignition flash points of new jet fuels, the role of ceiling architecture on detector and sprinkler response, and the ability of computer models to predict fires.

NIST Technical Note 1423 is available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402, (202) 512-1800. Order by stock number 003-003-03446-1. The report, a nine-minute video "Aircraft Hangar Fires: Fire Protection Improvements" and a CD titled "U.S. Navy High-Bay Hangar Tests" are available as a set from Joseph E. Gott, Naval Facilities Engineering Command, 299 Stovall Street, Code 150, Alexandria, VA 22332-2300, <jegott@hq.navfac.navy.mil>.

### IMPROVED WATTHOUR CALIBRATIONS SAVE TIME, MONEY

In response to its industry customers' needs, NIST now offers a new calibration service for watthour meters that provides faster turnaround time and reduced cost. Automation of a commercial calibrator and automated data collection/analysis software makes the improved service possible. What permits the full automation of the watthour meter calibration process is that only the three most requested test points (power factors of unity, 0.5 lag and 0.5 lead at set conditions of 120 V, 5 A and

60 Hz) are offered. The results: turnaround time cut to less than 2 weeks—approximately one-fourth of the time for standard watt-hour meter calibrations and the cost lowered to \$650—a greater than three times reduction in the fee.

The new service was developed after a quality effort by NIST identified repeated customer requests for these improvements.

For more information, contact James K. Olthoff, B344 Metrology Building, NIST, Gaithersburg, MD 20899-0001, fax: (301) 948-5796, <james.olthoff@nist.gov>.

## A GUIDE TO THE CONFORMITY ASSESSMENT MAZE

How can you be assured that the product you purchase will perform as expected? Conformity assessment procedures provide a means of ensuring that products, services or systems produced or operated in the United States do so. A new report by NIST, *The ABC's of the U.S. Conformity Assessment System* (NISTIR 6014), provides an overview of this nation's conformity assessment system for a better understanding of its impact on the marketplace.

The report describes the various steps in a conformity assessment system and the interrelationship between those steps. Included is discussion of standardization, inspection, testing, laboratory accreditation, and certification. In addition, management system assessment and registration (such as ISO 9000 and 14000 standards) are covered.

A printed copy of NISTIR 6014 may be requested by sending a self-addressed mailing label to Maureen A. Breitenberg, Building. 820, Room 282, NIST, Gaithersburg, Md. 20899-0001, fax: (301) 963-2871, <maureen.breitenberg@nist.gov>. The publication also can be obtained from the World Wide Web as an Adobe Acrobat file at <<http://ts.nist.gov/ts/htdocs/210/217/osc.htm>>. Multiple copies soon will be available from the National Technical Information Service, Springfield, VA 22161, (703) 487-4650, fax: (703) 321-8547, <orders@ntis.fedworld.gov>.

## NOVEMBER MEETING TO SPOTLIGHT SECONDARY LABS

The role of secondary laboratories in ionizing radiation measurements and standards will be the focus of the sixth annual meeting of the Council on Ionizing Radiation Measurements and Standards to be held at NIST headquarters in Gaithersburg, MD, Nov. 12-14, 1997. Secondary laboratories have been part of the network that maintains measurement standards in

ionizing radiation for more than two decades and are serving as a model for other U.S. calibration systems.

The technical program of the 1997 CIRMS meeting will cover medical applications, public and environmental radiation protection, occupational radiation protection, and radiation effects. CIRMS serves as a forum for discussing ionizing radiation measurements and standards issues, for identifying new needs of the user communities and for disseminating information on standards. CIRMS brings together representatives from academic, industrial and government agencies involved in nearly every aspect of ionizing radiation.

The registration fee is \$150. For registration information, contact Lori Phillips at B116 Administration Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-4513, fax: (301) 948-2067, <lori.phillips@nist.gov>. For technical information, contact Bert Coursey, C229 Radiation Physics Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-5584, fax: (301) 869-7682, <bert.coursey@nist.gov>.

## DON'T LET SALTS, MINERALS IN STEAM GET YOU BOILING!

It has been known for more than 50 years that depositions of solids—such as salts and minerals—from expanding steam can be a significant problem in the electrical power industry. In steam turbines, the impurities impair turbine efficiency by causing corrosion or constriction of passages. However, the same deposits (which have been only minor concerns for steam turbines) may be major headaches for the high-efficiency combustion turbines currently under development. The newer systems use steam from a steam turbine cycle to cool parts of a combustion turbine. Even small amounts of salts and minerals in the passages could lead to overheating and part failure.

To assist the power industry, NIST recently undertook a project to collect and, if possible, correlate all available data for the solubility in steam of various compounds. These compounds include sodium chloride, sodium hydroxide, sodium sulfate, sodium phosphate, iron oxides, copper oxides and silicon dioxide. The correlations will be helpful in developing steam purity recommendations for the conditions encountered in the advanced turbines.

The temperature range considered was from 200 °C to 900 °C (with 300 °C to 640 °C as the range of primary interest). Pressures considered were from 1 mPa to 6 mPa (with 2 mPa to 4 mPa as the range of primary interest). The data collected are contained in *Evaluation and Correlation of Steam Solubility Data for Salts and Minerals of Interest in the Power Industry* (NIST Technical Note 1387).

NIST TN 1387 may be ordered for \$7 from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402-9325. Order by stock number 003-003-03470-3.

#### **REPORT DETAILS WORKSHOP ON AMMONIA/WATER MIXTURES**

Can ammonia/water working fluids be used to replace steam in the design of some electrical power plants? Yes, but it is vitally important to know first the thermophysical properties of these mixtures. That was the conclusion of the Workshop on Thermophysical Properties of Ammonia/Water Mixtures held at NIST in Boulder, CO, in June 1996. The report from that workshop is now available.

Included is NIST's work investigating properties in the temperature range from 280 K to 450 K with a maximum pressure of 3.5 mPa and with ammonia mol fraction compositions in the range 0.8 to 1.0 for geothermal applications. The project involves the evaluation of literature data, experimental measurements, and the development of models for the thermodynamic and transport properties over these ranges. Industry interest, as learned from the workshop, is for properties up to 895 K at pressures to 22 mPa, and for the complete range of compositions.

Industry and government representatives at the workshop agreed to work together to investigate funding options, maintain communication among themselves, and to meet again, as appropriate, to reassess the status of properties work on the ammonia/water binary mixtures.

A copy of the *Report of the Workshop on Thermophysical Properties of Ammonia/Water Mixtures* (NISTIR 5059) is available for \$38 from the National Technical Information Service, Springfield, VA 22161, (703) 487-4650, <orders@ntis.fedworld.gov>. Order by PB 97-167522.

#### **APPROVAL/RECOGNITION OF ACCREDITATION BODIES**

The NIST Accreditation Body Evaluation Program (ABEP) is responsible for approving (recognizing) qualified domestic and foreign accreditation bodies that wish to engage in the accreditation of fastener testing laboratories under the Fastener Quality Act. The ABEP has been operating this program since publication of the final implementing regulations in September 1996. To date, three accreditation bodies have applied for approval/recognition. Their evaluations have been completed and the results presented to an "ABEP Panel," consisting of members from within and outside

NIST. The accreditation bodies approved by the ABEP Panel as of April 23, 1997, are: American Association for Laboratory Accreditation, National Aerospace and Defense Contractors, and United Kingdom Accreditation Service.

Effective April 23, 1997, and in accordance with the Fastener Quality Act, these organizations have been authorized to engage in the accreditation of laboratories that test fasteners and raw materials used in fastener manufacturing under the act. To be sold in the United States, all covered fasteners must be tested in an accredited laboratory after May 28, 1998.

#### **ENERGY-RELATED INVENTIONS PROGRAM MAKES RECOMMENDATIONS**

During the month of April, the NIST Office of Technology Innovation recommended three innovative technologies for commercialization to its Department of Energy partner under the Energy-Related Inventions Program.

High Throughput Manufacturing of High-Efficiency Solar Cells—a continuous vacuum process designed to replace conventional vacuum batch processes for manufacturing high efficiency CdTe solar cells for generating electricity; Novel Method and Apparatus for Ejecting Particulates from the Primary High Temperature Inlet Flow Path of a Coal Fired Turbine Generator—a device for removing, by centrifugal force, solid particulate matter from the high temperature exit stream of a coal gasifier; The Road Patcher—an assembly of hydraulic components designed to be installed easily on a conventional dump truck.

#### **SPECIAL NIST MEASUREMENTS AID MANUFACTURER OF SUPERCONDUCTING MAGNETIC ENERGY STORAGE SYSTEMS**

A series of electromechanical tests has been performed on a new aluminum stabilized coil structure by NIST scientists. The measurements were carried out at liquid-helium temperature in a 4 T magnetic field on a set of composite rings designed to simulate new magnets being constructed for superconducting magnetic energy storage (SMES) using high-purity (99.995 %) aluminum and a high-strength aluminum alloy (6061-T6). The rings were designed to simulate the mechanical and electrical characteristics of the aluminum stabilizer system designed for the Anchorage Municipal Light & Power SMES magnet by a private company. The conductor system is based on a conventional copper-stabilized niobium-titanium superconducting cable; however, for additional stability, the cable is encased in a high-purity aluminum jacket.

Stability in this sense refers to the provision of an alternative electrical conducting path and mechanical constraint should the superconductor revert to normal state. The increased stability is necessary because of the magnet's high energy-storage capacity, 1800 MJ. A high-strength aluminum-alloy overwrap is added to the conductor for structural support. Aluminum was selected instead of copper as the primary stabilizer material to reduce the magnet's size, mass, and cost while actually increasing its stability. The team adapted the division's unique superconducting composite ring measurement system to simulate operating conditions within the SMES magnet and, specifically, to test the effect of mechanical fatigue and magnetic field on the composite's resistivity (the conductor will be subjected to fatigue as the magnetic field is cycled in response to the power system's requirements).

The test results showed that the actual stabilizer degradation is much lower than predicted by a simplified analytical model. Ultimately, the private company and the expanding power utility industry will use these data to optimize the stabilizer design, substantially reducing the required quantity and cost of materials.

#### **NIST REPORT OUTLINING MEASUREMENT CHALLENGES FOR DEREGULATED U.S. ELECTRIC-POWER INDUSTRY DRAWS INTEREST, PRAISE**

A recently issued report, identifies the measurement challenges to be faced by the rapidly deregulating electric power industry and is drawing intense interest from industry and praise for its conception and implementation. The report was developed by NIST scientists in consultation with experts in industry, other government agencies, and universities.

Measurement Support for the U.S. Electric-Power Industry in the Era of Deregulation (NISTIR 6007) describes the changes taking place in the industry, translates those changes into key technical needs that industry must address, and describes the measurement capability required for a successful response. The technical needs of the electric-power industry are described in response to the principal driving forces of the electric-power industry: (1) efficiency, reliability, and stability of the power system; (2) equity in trade, and international competitiveness; (3) global warming and health effects; and (4) power quality. The study identifies the most critical long- and short-term measurement needs that require NIST's assistance, with focus on electrical measurements for the transmission and distribution of electric power. Finally, the document distinguishes between the measurement needs that NIST is currently able to address and those that remain to be addressed.

The U.S. electric-power industry is being deregulated to promote competition in providing electricity at lower cost and with greater responsiveness to customers' needs. This is a dramatic change for a huge industry that has annual electricity sales of \$208 billion (1995), employs 441 000 people (1995), and impacts the lives of every U.S. company and citizen. As a result of deregulation, more companies are likely to enter the business of providing electricity; both newcomers and existing providers will be seeking a competitive edge. Their individual success, and the collective benefits realized for the nation as a result of competition, will be highly dependent on their ability to adopt and exploit modern and emerging technologies. Exploiting these technologies effectively will require new measurement capability. NIST staff have looked into the future and produced a study that identifies the measurement capability required.

#### **CALCULATIONS OF MAGNETIC FIELD MEASUREMENT ERROR DISTRIBUTIONS EXPLAIN WHY INSTRUMENTS MAY DIFFER IN READINGS BY MORE THAN 30 %**

NIST scientists recently have calculated the probability distributions for the errors when three-axis and single-axis magnetic field meters are used to measure a dipole field, taken as a reasonable approximation of the fields produced by many appliances. The calculated results provide an explanation of why two identical three-axis magnetic field meters used to make measurements at the same point in space near an electrical appliance in the highly nonuniform field produced by the appliance can yield values that differ by more than 30 %. In each case, the distributions are found to be asymmetric. However, with knowledge of the 68 % and 95 % confidence intervals, determined as part of the computations, estimates of uncertainty can be assigned to measurements performed in fields having large gradients. The results of the calculations, presented in a tabulated format, also allow selections to be made of probe size which are consistent with total permitted measurement uncertainties.

The concern in the mid 1970s regarding health effects from exposure to electric and magnetic fields in the vicinity of power lines has shifted in recent years to health effect concerns from exposure to power frequency magnetic fields in residences, the work place, and in transportation systems. The magnetic fields in these environments can be highly nonuniform, particularly near electrical equipment such as motors, transformers, and heating elements. Because magnetic field measurements are frequently made with field meters employing circular coil probes, the measured field

values are actually averages over the cross sectional area of the probe. Since the measurement location is normally taken to be the center of the probe, differences between the average field and the central field can be regarded as a source of measurement error. Previously, calculations have been performed at NIST to determine the maximum error that could occur when magnetic field meters with single-axis and three-axis coil probes were used to measure the magnetic flux density produced by a small loop of alternating current, i.e., a magnetic dipole.

### **NIST RESPONDS TO INDUSTRY REQUESTS FOR MICROWAVE-FREQUENCY MATERIAL MEASUREMENTS**

NIST scientists responded to a number of industry requests for special measurements of the dielectric and magnetic properties of specimen materials at microwave frequencies. In one instance, a company wanted the information to enable them to choose absorber material for a radiofrequency anechoic chamber. This company supplied specimens of 13 different magnetic materials for characterization over the frequency range of 30 MHz to 2 GHz by means of a 14 mm-diameter coaxial transmission/reflection apparatus. These specimens also were measured with a NIST-developed permeameter to confirm the results from the coaxial measurements over the frequency range of 30 MHz to 100 MHz. In a second example, the company requested measurements of the complex permittivity of a glass dielectric material over the frequency range 8.2 GHz to 12.4 GHz (X-Band). A waveguide transmission/reflection method was employed. In a third instance, three companies requested a total of 75 measurements on various substrate materials over a frequency range of 1 GHz to 10 GHz. NIST scientists carried out these measurements using the split-post resonator technique specifically developed to provide industry with accurate and expedient measurements of printed wiring board and substrate materials.

### **ASSEMBLY LEVEL TOLERANCING WORKSHOP**

The Assembly Level Tolerancing: Standards and Implementation Issues workshop, was held April 24–25, 1997, at NIST. The workshop provided an excellent forum to: (1) identify and develop system level tolerancing standards and technology; (2) provide an open forum for CAD/CAT vendors and manufacturing industry to identify the gaps in the current available technology and to find some suitable solutions and alternatives; and (3) provide a roadmap that will aid in directing

future research, development, and support to bring about rapid development and technology transfer.

The workshop was well attended and included representation from industry end users, standards community, academia, computer aided design, and tolerancing software vendors. Topics discussed included tolerance theory and standards, representation and implementation, tolerance analysis, industrial applications and benchmarks, STEP implementation and advanced tolerancing methodologies (statistical and knowledge-based). The workshop proceedings will be made available as a NIST IR. Details of the workshop can be found at <http://www.nist.gov/mel/div826/event/workshop/assembly/workshop.htm>.

### **QUANTUM ENZYMOLOGY SUGGESTS ROUTE TO NEW ANTIBIOTIC DEVELOPMENT**

As part of a broad, long-range effort involving protein engineering and rational drug design, NIST and University of Maryland scientists collaborating at the Center for Advanced Research in Biotechnology have used new *ab initio* quantum mechanical techniques combined with high-resolution structural information from protein x-ray crystallography to propose a novel, microscopic chemical mechanism for the hydrolysis of  $\beta$ -lactam antibiotics by *Staphylococcus aureus* PC1  $\beta$ -lactamase. The therapeutic effectiveness of the penicillins and the cephalosporins has been reduced significantly by the emergence of resistant strains of bacteria that contain new enzymes able to break down these compounds before they can carry out their designed role. The limited, qualitative mechanistic information for this class of enzymes has been an obstacle to the development of next-generation penicillins and cephalosporins. This alternative mechanism suggests that the proper structural framework for substrate binding and orientation during catalysis plays a larger role in catalysis than previously appreciated. These insights provide new guidance to the pharmaceutical industry in the development of non- $\beta$ -lactam antibiotics to kill bacteria.

### **NEW METHODS FOR DNA DIAGNOSTICS**

Researchers at NIST are developing improved methods for DNA mutation detection using capillary electrophoresis (CE). Analysis of DNA by single strand conformational polymorphism (SSCP) provides an efficient means of screening these mutations before the costly and time-consuming task of sequencing is begun. SSCP is a method used to detect single-base changes (mutations) in DNA that introduce conformational changes in the polymer. The finding that these CE-SSCP measurements have specific temperature



dependencies provides the basis for developing this technique as an improved method for genetic profiling. For example, in the p53 system under study at NIST, specific mutations may be identified by CE-SSCP. (Mutations in the p53 gene are highly correlated to many forms of cancer.)

This work is being done in collaboration with a private company. Preliminary results were presented at the HUGO Meeting on Mutation Detection, May 29, 1997, in Brno, Czech Republic. Complementing these studies, computerized RNA folding analyses are being performed to predict which mutations are detectable by SSCP. In collaboration with scientists at a private company and the Laboratory of Experimental and Computational Biology of the National Cancer Institute, preliminary findings have resulted in molecular models of SSCP that are predictive. This information is being used in the design of measurement conditions and standards for SSCP.

#### UNDECOMPOSED HYDROGEN PEROXIDE ACCELERATES REACTION RATES IN SUPERCRITICAL WATER OXIDATION REACTORS

Recent experiments reveal the advantages of using hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) instead of oxygen ( $\text{O}_2$ ) as an oxidant in supercritical water oxidation (SCWO) flow reactors. The results obtained, resolve a long-standing inconsistency in the literature, establish some operational benefits, and provide insight into the mechanism of SCWO reactions.

Industrial SCWO systems effectively treat organic wastes by taking advantage of the high mutual solubility of organics and oxidants above the critical point of water (647 K, 22 MPa). Oxidation reactions typically achieve a high level of completion and generate benign effluent products (e.g., carbon dioxide and water). However, inconsistencies have been reported in the extent of reaction where  $\text{H}_2\text{O}_2$  has been used. In some studies,  $\text{H}_2\text{O}_2$  behaves identically with  $\text{O}_2$ , while in others it accelerates oxidation, and yet in others, it leads to large scatter in the data.

At NIST, experiments with  $\text{H}_2\text{O}_2$  oxidant are compared with tests where  $\text{O}_2$  is substituted (i.e., generated via peroxide thermolysis). The results show that careful decomposition of  $\text{H}_2\text{O}_2$  is the key. If  $\text{H}_2\text{O}_2$  is quantitatively thermolyzed to  $\text{O}_2$ , then mixed with the waste, literature results with  $\text{O}_2$  are reproduced. For reactions with undecomposed  $\text{H}_2\text{O}_2$ , accelerated rates are observed with ~260 % decrease in activation energy vs.  $\text{O}_2$ . For varying levels of decomposition, a range of reaction rates are observed. Thus, NIST researchers can reproduce the spectrum of literature data with  $\text{H}_2\text{O}_2$  and

attribute the variable behavior observed to incomplete decomposition to  $\text{O}_2$ . The researchers also have demonstrated that  $\text{H}_2\text{O}_2$  is more thermally stable under supercritical conditions than previously thought, which is reasonable since decomposition to  $\text{O}_2$  is thermodynamically suppressed at elevated pressures. The accelerated reaction rates with  $\text{H}_2\text{O}_2$  can offer substantial process advantages over pure  $\text{O}_2$  by allowing reactions to be conducted at lower temperatures ( $\Delta T \approx 50 \text{ K}$  to  $150 \text{ K}$ ) which reduces energy and material costs. Further, because the effective activation energies with  $\text{H}_2\text{O}_2$  are substantially lower, reactions are less sensitive to temperature excursions resulting in more robust operation. Finally, the results suggest that the OH radical is important in SCWO reactions, and that data from previous studies using  $\text{H}_2\text{O}_2$  must be interpreted carefully.

#### NIST MEASUREMENTS SUPPORT MORE ACCURATE $^{190}\text{Pt}$ HALF-LIFE DETERMINATION

A method developed recently at NIST for the determination of the platinum group elements (PGEs) in automobile catalysts has been applied to the analysis of unique geologic and meteoritic samples. Scientists at NIST have collaborated with a team from the University of Maryland and Colorado State University to study osmium and platinum in a set of 250 million year old rocks from Noril'sk, Siberia. This study resulted in a significantly more accurate determination of the half-life of  $^{190}\text{Pt}$ . The two elements are linked by the radiogenic system based on the alpha decay of  $^{190}\text{Pt}$  to  $^{186}\text{Os}$  and previous investigations were difficult because  $^{190}\text{Pt}$  is rare in nature. In addition, it has an extremely long half life, previously poorly defined as  $(650 \pm 46)$  billion years (95 % confidence limit) based on reported literature data ranging from 470 billion to 690 billion years.

The University of Maryland-Colorado State University team has expertise for high-precision isotopic measurements of osmium, but they did not have the comparable capability for platinum measurements.  $^{190}\text{Pt}$  in the Noril'sk rocks was determined at NIST using the isotope dilution inductively coupled plasma mass spectrometry method developed earlier for the determination of PGEs in used automobile catalyst materials. These measurements allowed the first precise isochron to be demonstrated for the Pt-Os isotopic system. The ratio of the natural concentrations of osmium and platinum plotted versus the amount of  $^{190}\text{Pt}$  daughter product ( $^{186}\text{Os}$ ) would normally be used to determine the age of rocks. However, in this experiment, rocks of well-known age were used to determine

the  $^{190}\text{Pt}$  decay constant as  $1.54 \times 10^{-12}$  per year with an uncertainty of about 1 %, which translates to a  $^{190}\text{Pt}$  half life of 449 billion years. In addition, the University of Maryland–Colorado State University team also has shown that these precise Pt and Os measurements can be used to probe interactions between the Earth's core and mantle and promises to be very useful in studying other fundamental geochemical processes.

#### **NEW INVENTION AIDS SEMICONDUCTOR MANUFACTURING**

Optical scattering is used in the semiconductor industry to measure microroughness and to detect particulate contaminants and subsurface defects on silicon wafers. As the feature sizes in modern integrated circuits continue to shrink, ever stricter demands are being made on instruments to detect smaller and smaller contaminant particles. One important issue that limits the sensitivity of such instruments is that light scattering due to the silicon wafer microroughness can obscure or overwhelm the scattering due to particles. This problem is widely recognized. The Semiconductor Industry Association roadmap declares that detection of particles with diameters less than  $0.1\text{ }\mu\text{m}$  is a potential “show stopper.”

Researchers at NIST have pursued an ingenious solution to this problem. By developing a novel technique, bidirectional ellipsometry, they found that the light scattered by microroughness has a characteristic, well-defined polarization for each scattering direction. Other scattering sources, such as particle contaminants and subsurface defects, scatter with different characteristic polarizations. The discovery of polarization signatures enabled the design of a microroughness-blind hemispherical optical scatter measuring instrument. A patent application has been filed on this invention, which addresses the issue of measuring nanoscale particles on silicon wafers; however, it is expected that bidirectional ellipsometry also will be a powerful technique for identifying and characterizing defects in optical components, data storage materials, and film coatings.

#### **TWENTY-FIFTH ANNIVERSARY CORM CONFERENCE**

The Council for Optical Radiation Measurements (CORM) is an industry organization that works with NIST to identify industry needs and to deploy new NIST services in its area of expertise. Founded in 1972, CORM has grown to represent about 150 companies in their search for a consensus on pressing problems and national needs in optical radiation measurement.

In recent years, CORM has held an annual meeting to conduct business and to hold workshops in specific areas of common concern. This year's annual meeting was held at NIST April 28–May 1, 1997, and focused on recent advances in optical remote sensing, reflectance and transmittance metrology, and infrared metrology. The program featured invited presentations by experts in government, academia, and industry.

#### **WORKSHOP ON NEEDS FOR COLOR AND APPEARANCE STANDARDS**

In conjunction with the CORM annual meeting, NIST participated in a workshop on April 28, 1997, to allow industry to comment on their needs for measurement standards in the fields of color and appearance. This CORM-organized workshop provided the attendees the opportunity to help define and prioritize ongoing NIST work. A previous workshop last year was sponsored by NIST to help researchers better understand industrial needs for color and appearance measurements and standards. Subsequently, NIST work was begun to advance the science and capability of appearance measurements.

The NIST objective is to develop measurement services and Standard Reference Materials for appearance attributes such as color, gloss, translucency, luster, sheen, texture, orange peel, distinctness of image, and contrast. These standards are most important to the paint, coating, and textile industries, and to the myriad of other industries that make consumer goods where specific color and appearance are quality attributes. This workshop helped to establish the desired physical properties and ranges of measurement quantities for these reference materials.

#### **WORKSHOP ON RETROREFLECTANCE MEASUREMENTS**

Also in conjunction with the CORM annual meeting, NIST held a workshop on May 1, 1997, to allow industry to comment on their needs for measurement standards in the field of retroreflectance. Retroreflectant materials are used widely and, in many cases required, to increase the visibility and conspicuity of people, vehicles, signs, and safety markings on the nation's highways and in transportation industries to reduce deaths and injuries.

Currently, there are no national measurement standards for retroreflective materials, and the workshop participants conveyed the need to have NIST provide such standards. The suggestions provided at this workshop will be used to formulate the NIST effort for retroreflective measurements.

### SHORT COURSE IN RADIATION THERMOMETRY

On May 6-8, 1997, NIST and the ASTM Committee E20.02 on Radiation Thermometry inaugurated a new Short Course on Temperature Measurement by Radiation Thermometry. It was designed by experts from NIST and a professor at Purdue University and editor of *Theory and Practice of Radiation Thermometry*. It was conducted in the new radiometric facility located in Building 245: FARCAL, the Facility for Advanced Radiometric Calibrations.

The course consisted of lectures that cover the fundamentals of radiometry and temperature measurements. They were complemented with hands-on, skill-building, problem-solving laboratory experiments. During these exercises, the participants learn ASTM voluntary industry standard test methods. They gain practical laboratory experience using commercial radiometers and blackbody sources that are loaned to the ASTM committee, and learn firsthand about the treatment of the measurement equation and proper uncertainty analysis.

The enrollment for the course was limited so that each laboratory instructor could concentrate on at most four students. Fourteen participants enrolled in and completed the course, including representatives from federal agencies such as NASA, the Navy, Los Alamos, and NIST, and industries located in Colorado, California, Oregon, and Ohio. Based on its success, it will be offered again next year.

### IMPROVED RUBIDIUM FREQUENCY STANDARD

A collaboration between NIST scientists in Boulder and guest researchers from both Switzerland and the People's Republic of China has produced a major improvement in the short-term stability of a rubidium-cell frequency standard. With the short-term stability now within a factor of three of the stability of commercial hydrogen masers, this new class of rubidium standards should meet the growing need for a higher performance local oscillator in the next generation of laser-cooled, primary frequency standards. Hydrogen masers also can meet this requirement, but they are much more complex and much more costly. These new frequency standards can be miniaturized and should be sufficiently low cost that they might find application in such areas as telecommunications synchronization.

This success is a result of several changes in design and careful attention to noise within the optics and the microwave frequency synthesizer. A key change is the use of a diode laser, rather than a discharge lamp, for

optically pumping the rubidium cell. This reduces noise associated with the broad spectrum of pump light generated by a discharge lamp. However, amplitude and frequency noise on the diode-laser output, and phase-modulation noise on the synthesized microwave probe, must be suppressed substantially to achieve the improved performance. The present stability of  $3 \times 10^{-13}$  at 1 s integrated time is an order of magnitude better than that of the best conventional rubidium-cell standards available today.

### DETECTING INTERNAL ELECTRIC FIELDS IN THIN FILMS USING SECOND HARMONIC GENERATION

Ferroelectric oxide thin films are being developed for integrated photonic devices such as modulators and switches for telecommunications and optical computing. Naturally occurring imperfections in the ferroelectric film, e.g., point defects or crystalline mismatches at interfaces, can induce internal electric fields that degrade the properties and reliability of the device. Measurement of these fields by electrical techniques requires that the film be sandwiched between electrodes, which is undesirable for photonic devices because conventional electrodes are not transparent.

NIST has developed an alternative optical technique for detecting these defect-induced fields in ferroelectric oxide films. The technique is based upon second harmonic generation, a phenomena that occurs in non-centrosymmetric materials whereby the material illuminated by laser light generates light at twice the fundamental frequency of the incident light. Internal electric fields introduce noncentrosymmetry into the material and thus generate second harmonic signals. In studies of ferroelectric barium titanate films, the fundamental frequency of the incident light was varied to measure independently second harmonic generation at the film/substrate interface, at the film/air interface, and in the bulk of the film. Results of these measurements have shown electric fields generated by film imperfections can be of comparable magnitude to the intrinsic fields generated by the ferroelectric nature of the film. This technique is used successfully to discriminate between ferroelectricity and imperfection-induced fields.

### NEW TECHNIQUE ALLOWS MECHANICAL PROPERTY MEASUREMENTS OF THIN FILMS USING SPECKLE INTERFEROMETRY

Electronic speckle pattern interferometry is a widely used experimental mechanics technique. Its big advantage is that no grating is needed on the specimen

surface. In the usual technique, two opposed laser beams interfere to create closely spaced lines of illumination. Fringes of displacement are generated by subtracting speckle pattern images taken at different strain levels. NIST scientists, when performing mechanical properties measurements on thin film samples, found that the density of speckles was too low to form measurable fringes. A new data reduction technique was developed to solve this problem. From a series of speckle images, a record of light intensity as a function of strain increment is formed for each pixel. These records are sinusoidal, and each cycle signifies an additional displacement of the order of the wavelength of light. A Fourier method is used to demodulate the data and calculate the total phase advance at each pixel. The result is an array of several thousand displacement values corresponding to pixel locations in the original images. This technique is used to study the mechanical behavior of thin films of metal and epitaxial silicon.

#### **NIST SCIENTIST SHARES JAPANESE PUBLICATION AWARD**

At the April meeting of the Japanese Society for Dental Materials and Devices, the society presented its annual award for the publication selected as the best that appeared in its two journals during the past year. For 1996, the award was presented to the head of the Department of Dental Engineering at Tokushima University, and a NIST scientist. The paper is entitled "Simulation of Transient Thermal Stress in Gypsum-bonded Investment," and it was published in the December 1996 issue of the *Dental Materials Journal*.

The paper provides insight into the development of stresses and strains within a mold as affected by material properties, temperature, mold heating rates, and external mold constraints. These variables can affect mold integrity and dimensions of cast appliances. To provide dental prostheses that fit a patient's mouth, shrinkage from the casting temperature is compensated for by the inclusion of refractory material that expands by phase transformation during the heating of the investment. Temperatures of about 700 °C are reached. The high temperatures and phase-induced expansions make it impractical to experimentally determine the stresses and strains throughout a dental mold. Simulation provides valuable understanding and predicting material and processing properties.

#### **NATURAL MAGNETIC MULTILAYERS DISCOVERED**

The BaO-Fe<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub> system offers the exciting possibilities of new materials that combine the high dielectric

properties of the barium titanates (e.g., BaTiO<sub>3</sub>) with the strong magnetic properties of the barium ferrites (e.g., BaFe<sub>12</sub>O<sub>19</sub>). In recent NIST research on phase equilibria in the BaO:Fe<sub>2</sub>O<sub>3</sub>:TiO<sub>2</sub> system, numerous complex ternary phases have been discovered. Most of the new compounds are of a new structure type and require complete structural determination. Recently, a group of the six compounds has been studied by high-resolution transmission electron microscopy. The structural modeling based on high-resolution imaging and computer simulations has led to the conclusion that there is a common structural architecture for all six phases. The structures are built out of two distinct two-dimensional blocks. One block, P, has a structure related to the barium titanates and, therefore, is expected to have dielectric properties. The thickness of this block (typically between 1.5 nm to 4 nm) can be controlled by the composition of the compound. The second block, H, has a structure related to the magnetic ferrites. The distribution of Fe is very inhomogeneous and mostly localized in the H block. Overall, the structures can be considered as materials with natural magnetic multilayers. With appropriate processing, a high degree of long-range order between the layers can be achieved. NIST researchers are now in the process of exploring possible useful properties of these new materials as well as understanding the physics of magnetic interaction between the pseudo-two-dimensional layers.

#### **LAVENT ADOPTED AS PART OF THE NEW EDITION OF NFPA 204 GUIDE FOR SMOKE AND HEAT VENTING**

At the recent annual meeting of the National Fire Protection Association (NFPA), the membership adopted a new edition of NFPA 204: Guide for Smoke and Heat Venting. The major feature of the new guide is the inclusion of the computer fire model LAVENT (Link Actuated VENTs). This model was developed at NIST during 1987-89 with support from the American Architectural Manufacturers Association Research Foundation as a design tool for sizing building smoke and heat vents. LAVENT was designed to simulate the environment and the response of sprinkler links in compartment fires with draft curtains and fusible-link-actuated ceiling vents. The adoption of the new 204 guide represents the first time that an NFPA guide or standard has ever included a fully documented computer fire model.

It is anticipated that the adoption of LAVENT in a national consensus standard will have a major impact on fire safety in that it will provide an accepted practice for the design of new venting systems and the

evaluation of existing systems at a level of detail and sophistication that was not possible previously.

### 3M AND NIST COLLABORATE ON FIRE SUPPRESSION STUDY

Under a Cooperative Research and Development Agreement with a private company, scientists at NIST are determining fire suppressant criteria for controlling fires in energized electrical equipment. Current standards presume that the needed concentration of fire suppressant will be the same as for non-electrical fires. Tests by the private company have shown that as much as twice the agent may be needed. NIST scientists have now conducted a series of tests to measure analogous ratios for preventing ignition of an ethylene-air flame by a metal foil strip maintained at a controlled temperature. This simulates the heated surface that would result from a continuously energized electrical component. For a given concentration of agent, the minimum foil temperature needed for ignition was determined. The agent concentration at which no autoignition was observed (at the high temperature limit of the foil) was the inerting concentration. These results then were compared to the agent concentration needed to suppress a small diffusion flame in the standard cup burner. The addition of two inert suppressants, either nitrogen or a commercial nitrogen-argon-carbon dioxide mixture, resulted in steady increases in the minimum ignition temperature up to the inerting level. On a volume basis, nitrogen was more effective than the mixture, requiring about 50 % more than that needed to extinguish the cup burner flame. Four halocarbon suppressants required about two to five times the cup burner extinguishment concentration. Two of these,  $C_4F_{10}$  and  $C_3HF_7$ , showed slight enhancement of ignition at sub-inerting concentrations. On a mass basis, all six suppressants required a factor of about one and a half to two times more than the cup burner value.

### S-CHECK, VERSION 2.0, AVAILABLE ON WORKSTATION

Clusters S-Check, NIST's sensitivity analysis tool for identifying performance bottlenecks in parallel programs, is now available on workstation clusters running PVM or MPI. The recent upgrade of the tool coincides with the emergence of cost-effective computing platforms using a cluster of workstations and off-the-shelf high performance interconnects. S-Check, Version 2.0, has successfully tuned a parallel application on NIST's experimental Asynchronous Transfer Mode cluster. NISTIR 6022, *S-Check, by Example*, demonstrates simple use of the tool. S-Check can be downloaded at <http://www.scheck.nist.gov/scheck>.

### TIMING ANALYSIS FOR FLAT PANEL DISPLAY INTERFACE (FPDI) STANDARD

At the request of the Video Electronics Standards Association (VESA), NIST analyzed flat-panel display interface timing characteristics, based on proprietary product information of manufacturers. NIST was trusted as an impartial participant to whom manufacturers sent panel specification sheets. The necessary timing characteristics were derived, converted to a uniform nomenclature, and returned to VESA with model numbers and proprietary information omitted. This information will be used as the technical foundation for a new interface timing standard for FPDI-2.

Flat-panel displays are an essential enabling technology for portable computers, desktop monitors, and instruments. Panel architecture makes proper timing much more critical than for cathode ray tube (CRT) displays; failure to meet the timing requirements can cause permanent damage to the panel. The standard timings and "plug and play" compatibility of CRTs have not been available for flat-panel displays, an omission that VESA is moving to correct.

VESA, an industry standards association and approved standards submitter, has developed display-related voluntary industry standards. NIST has played a significant role in the development of several recent VESA standards.

### SOFTWARE UNIFIES "SCATTERED" DATA FOR CHIP MAKERS

The ability to characterize how a silicon wafer scatters light—or how a light beam spreads out after hitting a wafer surface—can provide invaluable information for the successful manufacture of high-quality semiconductor chips. Unfortunately, optical scatter instruments lack standardization, making it difficult to compare values obtained by devices from different companies. A new NIST software package soon will offer chip makers a way to reconcile and compare data derived from different systems.

The NIST software uses an algorithm that compensates for the unique characteristics of individual optical scatter instruments and yields readings free of any variance. The purified data can be obtained for silicon wafers exhibiting microroughness. The American Society for Testing and Materials is currently incorporating this method into documents describing wafer inspection systems. The software is expected to become available by the end of August 1997.

For more information, contact Thomas A. Germer, A320 Metrology Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-2876, <[thomas.germer@nist.gov](mailto:thomas.germer@nist.gov)>.

## NEW STANDARD CALIBRATES FIBER MEASUREMENT SYSTEMS

Optical fiber chromatic dispersion is an important parameter that affects pulse broadening—and therefore the bandwidth—of lightwave communication systems. New undersea systems, which may be several kilometers in length, typically have an operational wavelength very close to the fiber's zero dispersion wavelength, challenging the accuracy capabilities of current measurement systems. These systems require a calibration standard with an uncertainty for zero dispersion wavelength in the 0.1 nm to 0.2 nm range.

In response to a request from the Telecommunications Industry Association, NIST developed Standard Reference Material 2524, which calibrates optical fiber dispersion measurement systems at an uncertainty level of  $\pm 0.06$  nm. Each unit is individually certified and consists of a 10 km-long dispersion-shifted optical fiber housed in an aluminum box (insulated with foam for thermal stability). Two bulkhead FC/PC type adapters are mounted on the front panel of the aluminum box for optical connection with the measurement system to be calibrated.

SRM 2524 can be purchased for \$3108 from NIST's Standard Reference Materials Program, Building 202, Room 204, Gaithersburg, MD 20899-0001, (301) 975-6776, fax: (301) 948-3730, <srminfo@enh.nist.gov>.

## VRML TEST SUITE HELPS BUILD VIRTUAL WORLDS

More and more developers of interactive three-dimensional graphics for the World Wide Web and virtual world builders are incorporating the Virtual Reality Modeling Language (VRML) specification into their products. To help check compliance with this emerging standard, NIST is working with the VRML Consortium, which consists of VRML experts, developers, and virtual world builders, to develop specific testing strategies and associated test suites.

NIST's VRML Test Suite (VTS) interprets the specification using a web-accessible system that provides the VRML specification, functional requirements, VRML test worlds, and expected results. The NIST VTS system can be used to test a browser's ability to implement various aspects of the VRML specification. Broad use of such test suites promotes interoperability among the various browsers. Both developers and users of VRML browsers can use the NIST VTS system to determine whether products handle shapes, colors,

texture, and movement through the virtual world in accordance with the VRML specification.

Several companies developing VRML browsers already have indicated that following NIST's tests will help them build higher quality products. NIST also is developing a VRML parser, which it expects to release in early August, to help virtual world builders to verify their virtual landscapes. For more information about NIST's VRML test suite, check out <<http://www.nist.gov/vrml.html>> on the World Wide Web or contact Mary Brady, 562 NIST North, NIST, Gaithersburg, MD 20899-0001, (301) 975-4094, <mary.brady@nist.gov>.

## MACHINE TOOL RESEARCH IN THE "FAST LANE"

Manufacturing is switching into the fast lane. From the aerospace industry to the automotive sector and beyond, companies are turning to high-speed machining, and for good reason. Faster machining speeds can translate into shorter cycle times, fewer parts, higher accuracy, better finishes, and other improvements.

Faster may indeed be better, but as spindle speeds and feed rates increase, experience quickly becomes an unreliable guide. A new NIST research testbed intends to help put high-speed machining on a more solid technical foundation. Studies of cutting dynamics, spindle performance, cutting tool designs and other topics will be conducted on a high-speed milling machine. It features an 18.5 kW spindle that is capable of speeds of more than 20 000 revolutions per minute and slides that travel 0.6 m/s. While some commercially available machine tools operate at even higher rates, spindle speeds on conventional machining centers, in contrast, are in the range of about 6000 rpm.

NIST's initial collaborators include researchers from two universities and a private company. Among projects already under way at the new testbed is a study of the vibrational characteristics of long cutting tools. With a specialized test fixture attached to the machine spindle, researchers will evaluate conditions during the transition from stable to unstable cutting behavior. This study and others will help extend a NIST computer model of milling processes to encompass high-speed machining. One aim is to reliably predict cutting performance on different materials and under various conditions.

Organizations interested in pursuing research at the new NIST testbed should contact Matt Davies, B106 Sound Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-3521, <mdavies@nist.gov>.

## POLARIZED SIGNATURES MAY LEAD TO TINIER CHIPS

Optical scattering is used by the semiconductor industry to measure the microroughness of a silicon wafer as well as detect particulate contaminants and subsurface defects. However, the ability of optical scattering instruments to do the job is limited by problems with sensitivity. Light scattering due to microroughness can obscure or overwhelm the scattering caused by particles.

NIST researchers have discovered a way to cleanly distinguish the scattering caused by microroughness from that of particulate contamination or subsurface defects. Using a novel technique called bidirectional ellipsometry, scientists at NIST found that light scattered by microroughness has a characteristic, well-defined, polarization “signature.” They also learned that other sources of scatter, such as particulate contaminants and subsurface defects, scatter light with unique polarizations different than those exhibited by microroughness.

With this knowledge, the researchers designed an instrument that is blind to the microroughness optical scattering pattern. Therefore, nanoscale particles on silicon wafers can be detected and measured without the former problem of interference. A provisional patent has been filed on the device.

What this means to the semiconductor industry is that particles with diameters less than 0.1  $\mu\text{m}$  soon may be routinely discernible. The Semiconductor Industry of America roadmap for the future declared that such ability would remove a major impediment toward the manufacture of tinier integrated circuits.

In addition to its usefulness in process inspection of silicon wafers, bidirectional ellipsometry is expected to become a powerful technique for identifying and characterizing defects in optical components, disk storage materials and film coatings.

For more information, contact Thomas A. Germer, A320 Metrology Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-2876, <thomas.germer@nist.gov>.

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## Standard Reference Materials

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**STANDARD REFERENCE MATERIAL® 956A, ELECTROLYTES IN FROZEN HUMAN SERUM**  
SRM 956a is intended primarily for use in the calibration and standardization of procedures employed in clinical analysis for the determination of specific

electrolytes in either diluted or undiluted human serum or plasma. This SRM can be used for standardizing direct reading ion-selective electrode analyzers and for validating working or secondary reference materials. The elements certified in SRM 956a represent some of the principal elements required by the human body to regulate muscle irritability (including the heart), hydration, regulation of cellular functions, protein synthesis, and a host of other functions. The ability of clinical laboratories and physicians to measure accurately these elements in serum plays a crucial role in the diagnosis and treatment of a variety of diseases and abnormalities as well as the maintenance of good health.

The certified concentrations of the serum analytes, calcium, lithium, magnesium, potassium, and sodium are the means of results based on measurements using a single primary method or two or more independent reliable methods. A unit of SRM 956a consists of six flame-sealed ampules of frozen human serum, two ampules each of three different concentration levels. Each ampule contains  $(2.00 \pm 0.04)$  mL of human serum.

## STANDARD REFERENCE MATERIAL® 1747, TIN FREEZING POINT CELL AND STANDARD REFERENCE MATERIAL® 1748, ZINC FREEZING POINT CELL

As part of its responsibility for the realization, maintenance, and dissemination of the International Temperature Scale of 1990 (ITS-90) for the U.S. industrial and scientific communities, NIST has developed and certified two additional SRMs for this purpose: Tin Freezing-Point Cell (SRM 1747) and Zinc Freezing-Point Cell (SRM 1748). The two freezing points required to realize ITS-90 from 0 °C to 420 °C are that of tin (231.928 °C) and zinc (419.527 °C). These freezing points are realized by using thermometric fixed-point cells containing high purity ( $\geq 99.9999$  % pure) metals. Tin and zinc freezing-point cells, with the triple point of water (TPW) cell (0.01 °C), are used for the ITS-90 calibration of standard platinum resistance thermometers (SPRTs) from 0 °C to 419.527 °C, for part of the calibration of SPRTs from 0 °C to 660.323 °C, and for part of the calibration of high-temperature SPRTs (HTSPRTS) from 0 °C to 961.78 °C.

SRM 1747 consists of approximately 1071 g of tin with impurities of 0.4 mg/kg; SRM 1748 consists of approximately 1031 g of zinc with impurities of 0.3 mg/kg. Both are contained in a high purity graphite crucible containing a high purity graphite re-entrant well.

**STANDARD REFERENCE MATERIAL® 965,  
GLUCOSE IN FROZEN HUMAN SERUM**

SRM 965 is intended primarily for use in evaluating the accuracy of procedures for the determination of serum glucose. The ability of clinical laboratories to measure accurately glucose in patient samples has a direct effect on the diagnosis and treatment of diabetes mellitus, a chronic disorder characterized by insulin deficiency, hyperglycemia, and high risk for development of complications of the eyes, kidneys, peripheral nerves, heart, and blood vessels. The disease is highly prevalent, affecting as many as 16 million people in the United States. The American Diabetes Association estimated in 1992 the direct medical costs associated with diagnosis and treatment of diabetes at \$45.2 billion. The indirect costs of diabetes, such as lost productivity and premature death, equal \$46.6 billion.

The availability of SRM 965 should assist the manufacturers of secondary standards and controls to value-assign more accurate values to their materials and thus reduce the costs associated with larger uncertainties due to matrix biases that are known to exist in freeze-dried controls. The overall impact of better monitoring of glucose is a cost savings to the clinical diagnostics community and the diabetes patient, thus ensuring a better quality of life and longevity.

A unit of SRM 965 consists of six flame-sealed ampules of frozen human serum, two ampules for each of three different glucose concentration levels.



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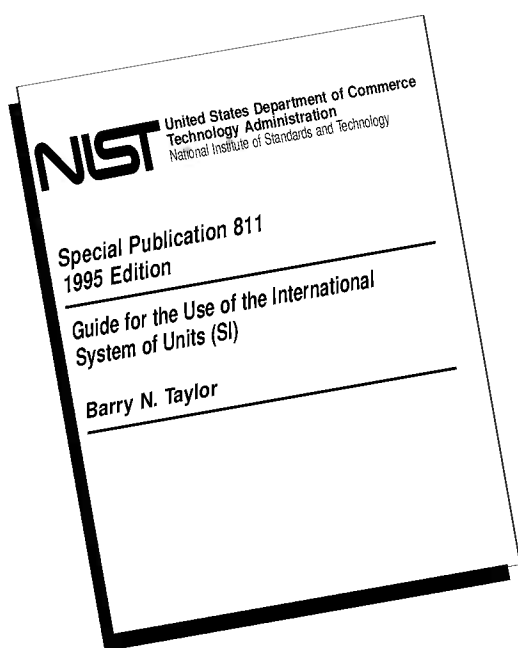
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# The International System of Units (SI)

## *The Modern Metric System*



Uncertain about the International System of Units (universally abbreviated SI), the modern metric system used throughout the world? Do you need to know the proper way to express the results of measurements and the values of quantities in units of the SI? Do you need to know the NIST policy on the use of the SI? Then you need the 1995 edition of the National Institute of Standards and Technology Special Publication 811, *Guide for the Use of the International System of Units (SI)*.

The 1995 edition of the National Institute of Standards and Technology Special Publication 811, *Guide for the Use of the International System of Units (SI)*, by Barry N. Taylor, is now available.

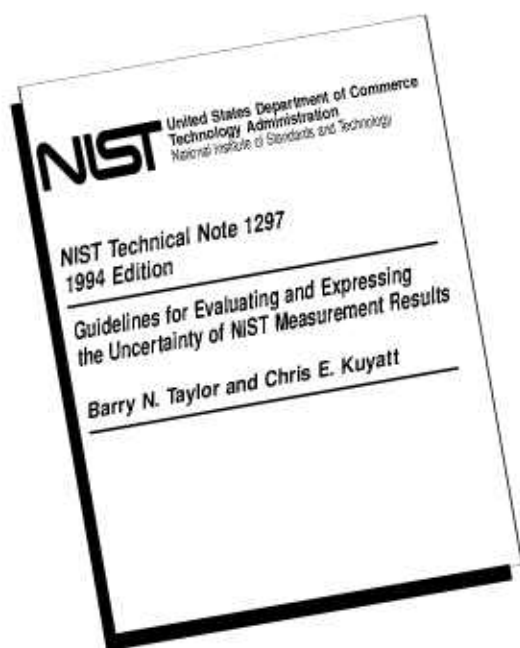
The 1995 edition of SP 811 corrects a number of misprints in the 1991 edition, incorporates a significant amount of additional material intended to answer frequently asked questions concerning the SI and SI usage, and updates the bibliography. The added material includes a check list for reviewing the consistency of written documents with the SI. Some changes in format have also been made in an attempt to improve the ease of use of SP 811.

The topics covered by SP 811 include:

- NIST policy on the use of the SI in NIST publications.
- Classes of SI units, those SI derived units that have special names and symbols, and the SI prefixes that are used to form decimal multiples and submultiples of SI units.
- Those units outside the SI that may be used with the SI and those that may not.
- Rules and style conventions for printing and using quantity symbols, unit symbols, and prefix symbols, and for spelling unit names.
- Rules and style conventions for expressing the results of measurements and the values of quantities.
- Definitions of the SI base units.
- Conversion factors for converting values of quantities expressed in units that are mainly unacceptable for use with the SI to values expressed mainly in units of the SI.
- Rounding numbers and rounding converted numerical values of quantities.

Single copies of the 84-page SP 811 may be obtained from the NIST Calibration Program, Building 820, Room 232, Gaithersburg, MD 20899-0001, telephone: 301-975-2002, fax: 301-948-3825.

# Evaluating and Expressing the Uncertainty of Measurement Results



Uncertain about expressing measurement uncertainty? Do you need to know how NIST states the uncertainty of its measurement results and how you can implement their internationally accepted method in your own laboratory? Then you need the newly available 1994 edition of the National Institute of Standards and Technology Technical Note 1297, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*.

The 1994 edition of the National Institute of Standards and Technology Technical Note 1297, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, by Barry N. Taylor and Chris E. Kuyatt is now available.

The 1994 edition of TN 1297 includes a new appendix—Appendix D—which clarifies and gives additional guidance on a number of topics related to measurement uncertainty, including the use of certain terms such as accuracy and precision. Very minor word changes have also been made in a few portions of the text of the 1993 edition in order to recognize the official publication in October 1993 by the International Organization for Standardization (ISO) of the *Guide to the Expression of Uncertainty in Measurement* on which TN 1297 is based. However, the NIST policy on measurement uncertainty, Statements of Uncertainty Associated with Measurement Results, which is reproduced as Appendix C of TN 1297, is unchanged.

It is expected that the 1994 edition of TN 1297 will be even more useful than its immediate predecessor, the 1993 edition, of which 10 000 copies were distributed worldwide.

Those United States readers who wish to delve into the subject of measurement uncertainty in greater depth may purchase a copy of the 100-page ISO *Guide* from the Sales Department of the American National Standards Institute (ANSI), 105-111 South State Street, Hackensack, NJ 07601. Copies may also be purchased from the ISO Central Secretariat, 1 rue de Varembe, Case postale 56, CH-1211 Genève 20, Switzerland.

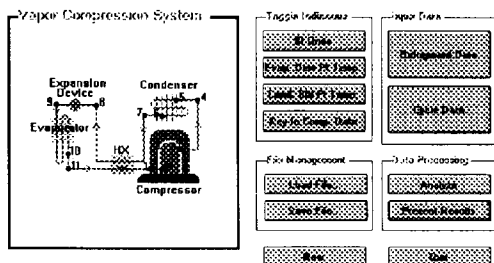
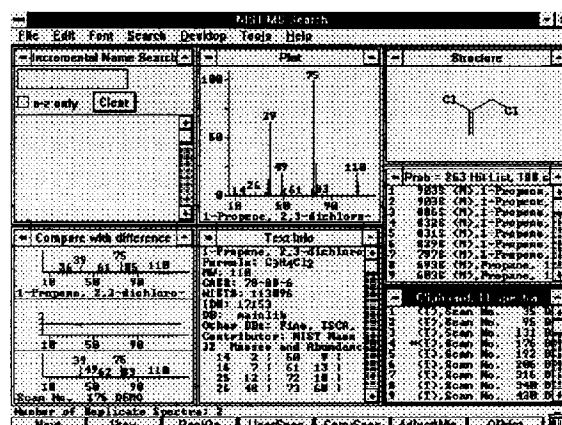
Single copies of the 20-page TN 1297 may be obtained from the NIST Calibration Program, Building 820, Room 232, Gaithersburg, MD 20899-0001, telephone: 301-975-2002, fax: 301-948-3825.



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